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Hop Extract

RECORDED

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PROVISIONAL SPECIFICATION.

[Communicated from abroad by Gustav Noback, Engineer, and Doctor Wilhelm Ginil, Professor, both of Prague, Bohemia, Austrian Empire.]

Modes of Treating and Utilizing Certain Waste Products Obtained in Brewing and similar Processes.

I JOHN CLAYTON MEWBURN, of 169 Fleet Street in the City of London, Fellow of the Institute of Patent Agents do hereby declare the nature of my invention for "MODES OF TREATING AND UTILIZING CERTAIN WASTE PRODUCTS OBTAINED IN BREWING AND SIMILAR PROCESSES," to be as follows:—

- 5 This invention consists mainly of a process or processes for recovering or obtaining in a simple manner the bitter matters and the resinous matters of hops contained to a large extent in the "rocks" of the fermenting casks or tuns or working squares as well as in the deposits or sediments in cooling vessels and store casks and in the deposits or sediments of yeast and also in the used or boiled hops.
- 10 The invention also comprises a process or processes for producing these bitter matters and resinous matters of hops in such forms as will allow of the direct use of these products in brewing or similar processes.

For the purpose of recovering or obtaining the above mentioned matters, the foam or froth of "rocks" or of boiled hops obtained by collecting it in a filter bag 15 or such like device, and also the deposits or sediments of yeast obtained in a similar manner, and the deposits or sediments in the cooling vessels and in the store casks collected in a filter bag, and the hops boiled or used in the process of brewing, after having allowed all liquor to drain or drop off from them are dried by the influence of the atmosphere or by artificial heat; the dry substance thus 20 obtained, after having been reduced in size by a grinding or reducing process is subjected to extraction by alcohol of high standard. It is recommended to accelerate or support this extraction by the employment of heat and to use for this process of extraction any of the well known apparatus, which are employed for the extraction of vegetable substances by alcohol at a raised degree of temperature 25 and with as little loss of alcohol as possible. The alcoholic extract of the said substances thus obtained is allowed to cool and to settle and is filtered if necessary and the alcohol is then distilled off in a distilling vessel or still which is strongly chloroform.

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tinned inside. Next a solution of carbonate of soda and water of a suitable degree of concentration or strength is poured on the residuum in the distilling vessel and the mixture is allowed to boil for a certain period. By this operation the greater part of the residuum is dissolved, and appears as a brownish liquor, and another part remains undissolved.

This latter part, after cooling of the liquor, is separated from the solution by filtration and is immediately subjected to a second boiling for a certain period; the decoction or product thus obtained, which is also of a brownish tint, is again separated from its residuum by filtration and this residuum, if necessary or desirous, may for a third time and even oftener be boiled with a solution of carbonate of 5 soda and then with pure water and finally separated by filtration or by decanting from the boiled liquor, and is dried in a drying apparatus or kiln at a temperature of 10 212° to 230° Fahrenheit or by the influence of the atmosphere. This final residuum contains or forms the mixture of the hop resins.

Of the brownish solutions obtained in the repeated process of boiling the 15 residuum from the distilling process, the more concentrated ones of a darker shade are used for obtaining the hop bitter substance, while the more diluted solutions obtained by the later or consecutive boilings may be employed for the boiling of fresh quantities. The said concentrated solutions, after having perfectly cooled down, are filtered to retain the small quantity of hop resin contained therein and 20 the solution is then carefully neutralized by pure muriatic acid.

By this neutralizing process a precipitate of a slight yellowish brown tint is obtained, which for its greater part consists of hop bitter. This precipitate is filtered and washed with clean water. The precipitate may be subjected to different processes for converting it into the proper form for use; it may be dissolved in 25 pure alcohol of high standard, in order to obtain an alcoholic solution of hop bitter; or it may be boiled with a 3 per cent solution of pure phosphate of soda and distilled water and thereby converted into an aqueous alkaline solution of the hop bitter matter; or it may be converted into syrup of sugar or into solid sugar, by mixing a hot saturated alcoholic solution of the precipitate with hot syrup of grape 30 sugar or hot syrup of invert sugar or with hot extract of malt and by concentrating the mixture until the alcoholic vapors escape. This mixture in the form of a syrup or of solid sugar, if dissolved in water, will give a sugar solution in which the hop bitter matter is divided in a fine emulsion.

The solution or emulsion of hop bitter matters obtained in one of the forms 35 above specified, may be used to partly replace the hops for the preparation of the boiled wort and also to increase the power of the beer in foaming. The solution or emulsion may for these purposes either be added to the boiling wort with the hops or as a part of the hops and be boiled with the wort or may be added to the wort after the boiling process of the latter, before the wort is transferred to the cooling 40 vessels. The proportion in which this hop bitter matter is added to the wort, will depend upon the quantity of hops which it is to replace and upon the amount of hop bitter in the solution or emulsion employed, which solution or emulsion may for the different purposes be produced in different degrees of concentration and may generally be of such form that 10 grammes of the hop bitter solution 45 or 50 grammes of the hop bitter sugar in the solid form, will correspond to 1000 grammes of hops.

The employment of these preparations will effect a considerable saving in hops, while preserving the same degree and quality of bitterness in the beer and does not in any way injuriously affect the quality of the beer.

The mixture of hop resin obtained or resulting from the process above specified, may be used (besides its use in boiling beer) for preparing a beer varnish for varnishing the cooling vessels and also the casks for storing and transporting beer. The mixture of hop resin for this purpose is dissolved in pure alcohol with an addition of about 5 per cent of ether and a varnish may thus be obtained of the 55 consistency or density required.

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As a modification of the foregoing process or processes for treating the waste products in order to recover the resinous and bitter matters of hops the following process may be employed :—

5 The said waste products are extracted with a 5 per cent solution of carbonate of soda in water first without heating and then with the employment of heat and the solution thus obtained, after being filtered, is precipitated with muriatic acid ; then the washed and dried precipitate is heated with alcohol or with chloroform or benzol whereby the bitter matters are freed from any admixtures so that after such purification the precipitate may be brought into one of the forms above specified.

10 That residuum or part obtained or remaining after the treatment of the waste products with carbonate of soda, may after washing with water and drying be boiled with alcohol and thus a solution of hop resin be directly obtained, which after an appropriate concentration may either be employed in boiling the wort or may be used for preparing a beer varnish.

15 Instead of carbonate of soda, I may also employ carbonate of potassa or carbonate of ammonia or the bi-carbonates of these alkalies or diluted solutions of caustic potassa or caustic soda. Again basic phosphates of soda as well as alkaline liquors in general are suitable for the purpose. I can also use these alkalies viz : carbonate of soda, carbonate of potassa, carbonate of ammonia, the double acidous salts of 20 these alkalies or caustic alkalies, instead of the phosphate of soda for the preparation of the solution of the purified hop bitter matters.

Dated this 21st day of May 1885.

J. C. MEWBURN.

COMPLETE SPECIFICATION.

**Modes of Treating and Utilizing Certain Waste Products
Obtained in Brewing and similar Processes.**

I JOHN CLAYTON MEWBURN of 169 Fleet Street in the City of London, Fellow of the Institute of Patent Agents do hereby declare the nature of this invention (a communication from abroad by Gustav Noback Engineer and Doctor Wilhelm Gintl, both of Prague, Bohemia, Austrian Empire) and in what manner the same is to be performed, to be particularly described and ascertained in and by the 5 following statement:—

This invention consists mainly of a process or processes for recovering or obtaining in a simple manner the bitter matters and the resinous matters of hops contained to a large extent in the "rocks" of the fermenting casks or tuns or working squares, as well as in the deposits or sediments in cooling vessels and storage casks and in the deposits or sediments of yeast; also in used or boiled hops and in the residuum from the process of extracting hops. The invention also comprises a process or processes for producing these bitter matters and resinous matters of hops in such forms as will allow of their direct use in brewing or similar processes. 10

For the purpose of recovering or obtaining the above mentioned matters, the foam or froth of "rocks" or of boiled hops obtained by collecting it in a filter bag or such like device and also the deposits or sediments of yeast obtained in a similar manner, and the deposits or sediments in the cooling vessels and in the storage casks, collected in a filter bag and the hops boiled or used in the process of brewing, 15 or the residuum from the process of extracting hops after having allowed all liquor to drain or drop off from them, are dried by the influence of the atmosphere or by artificial heat; the dry substance thus obtained, after having been reduced in size by a grinding or reducing process, is subjected to extraction by alcohol of high standard. It is recommended to accelerate or support this extraction by the 20 employment of heat and to use for this process of extraction any of the well known apparatus which are employed for the extraction of vegetable substances by alcohol at a raised degree of temperature and with as little loss of alcohol as possible. The alcoholic extract of the said substances thus obtained is allowed to cool and to settle and is filtered if necessary and the alcohol is then distilled off in 25 a distilling vessel or still, which is strongly tinned inside. Next, a solution of carbonate of soda and water of a suitable degree of concentration or strength is poured on the residuum in the distilling vessel and the mixture is allowed to boil for a certain period. By this operation the greater part of the residuum is dissolved 30 and appears as a brownish liquor, and another part remains undissolved.

This latter part, after cooling of the liquor, is separated from the solution by filtration and is immediately subjected to a second boiling for a certain period; the decoction or product thus obtained, which is also of a brownish tint, is again separated from its residuum by filtration and this residuum if necessary or desirable may for a third time and even oftener be boiled with a solution of carbonate of 35 soda and then with pure water and is finally separated by filtration or by decanting. 40

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from the boiled liquor and is dried in a drying apparatus or kiln at a temperature of about 21²° to 230° Fahrenheit, or by the influence of the atmosphere. This final residuum contains or constitutes the mixture of hop resin.

From the brownish solutions obtained in the repeated process of boiling the 5 residuum from the distilling process, the more concentrated ones of a darker shade are used for obtaining the hop bitter substance while the more dilute solutions obtained by the later or consecutive boilings may be employed for the boiling of fresh quantities. The said concentrated solutions after having become quite cool are filtered in order to separate the small quantity of hop resin contained therein 10 and the solution is then carefully neutralized by pure muriatic acid, or by other dilute mineral acid, such for example as sulphuric acid or by stronger organic acids, such as tartaric acid or acetic acid for example.

By this neutralizing process a precipitate of a slight yellowish brown tint is obtained, which chiefly consists of hop bitter. This precipitate is filtered and 15 washed with clean water. The precipitate may be subjected to different processes for converting it into the proper form for use; it may be dissolved in pure alcohol of high standard in order to obtain an alcoholic solution of hop bitter; or it may be boiled with a three per cent solution of pure phosphate of soda and distilled water and thereby be converted into an aqueous alkaline solution of the hop bitter 20 matter; or it may be converted into sugar syrup or into solid sugar, by mixing a hot saturated alcoholic solution of the precipitate with hot grape sugar syrup or hot invert sugar syrup or with hot extract of malt and by concentrating the mixture until the alcoholic vapors escape. This mixture in the form of a sugar syrup or of solid sugar, if dissolved in water will give a saccharine or sugar 25 solution in which the hop bitter matter is held or contained in the form of a fine emulsion.

The solution or emulsion of hop bitter matters obtained in one of the forms above specified, may be used to partly replace the hops for the preparation of boiled wort and also to increase the power of the beer in "foaming." The solution or 30 emulsion may for these purposes either be added to the boiling wort with the hops or as a part of the hops and be boiled with the wort or may be added to the wort after the boiling process of the latter and before the wort is transferred to the cooling vessels. The proportion in which this hop bitter matter is added to the wort will depend upon the quantity of hops which it is to replace and upon 35 the amount of hop bitter in the solution or emulsion employed, which solution or emulsion may for the different purposes be produced in different degrees of concentration and may generally be of such form that ten grammes of the hop bitter solution or fifty grammes of the hop bitter sugar in solid form will correspond to a thousand grammes of hops.

40 The employment of these preparations will effect a considerable saving in hops while preserving the same degree and quality of bitterness in the beer and does not in any way injuriously affect the quality of the beer.

The mixture of hop resin obtained or resulting from the process above specified may be used (besides its use in boiling beer) for preparing a beer varnish for 45 varnishing the cooling vessels and also the casks for storing and transporting beer. The mixture of hop resin for this purpose is dissolved in pure alcohol with an addition of about five per cent of ether and a varnish may thus be obtained of the consistency or density required.

As a modification of the foregoing process or processes for treating the waste 50 products in order to recover or obtain the resinous and bitter matters of hops, the following process may be employed.

The said waste products are extracted with a 5 per cent solution of carbonate of soda in water, first without heating and then with the employment of heat and the solution thus obtained after being filtered is precipitated by muriatic acid or other 55 dilute mineral acid, such as sulphuric or by stronger organic acids such as tartaric and acetic; then the washed and dried precipitate is treated with alcohol or with chloroform or wood spirit, sulphuret of carbon, petroleum-ether, or the volatile

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matters obtained in the rectification of petroleum, or with benzol or the volatile oils of the nature of turpentine whereby the bitter matters are freed from any admixtures, so that after such purification the precipitate may be brought into one of the forms above specified. That residuum or part obtained or remaining after the treatment of the waste products with carbonate of soda, may after washing 5 with water and drying be boiled with alcohol or with the solvents above named such as wood spirit, petroleum-ether, turpentine-oil &c and thus a solution of hop resin be directly obtained, which after an appropriate concentration may either be employed in boiling wort or be used for preparing a beer varnish.

Instead of carbonate of soda I may also employ carbonate of potassa or carbonate 10 of ammonia, or the bicarbonates of these alkalies or dilute solutions of caustic potassa or caustic soda. Again basic phosphates of soda as well as alkaline liquors in general are suitable for the purpose. I can also use these alkalies viz:— carbonate of soda, carbonate of potassa, carbonate of ammonia, the double acidous salts of these alkalies or caustic alkalies instead of the phosphate of soda for the 15 preparation of the solution of the purified hop bitter matters.

According to another modification of the process or processes hereinbefore described for treating the said waste products in order to recover or obtain the resinous and bitter matters of hops, the waste products are as before freed from liquor and dried by exposure to the atmosphere or by artificial heat; they are then 20 treated with wood spirit, sulphuret of carbon, petroleum-ether or the volatile matters obtained in the rectification of petroleum and such like mineral oils or with benzole or volatile oils of the nature of turpentine, whereby the resinous and bitter matters are extracted.

Having now particularly described and ascertained the nature of my said 25 invention and in what manner the same is to be performed as communicated to me, I declare that what I claim is

1. The several processes hereinbefore described of treating the waste products referred to whereby the bitter matters and resinous matters contained therein are recovered or obtained, substantially as set forth. 30
2. The treatment, substantially as set forth, of the precipitate obtained as hereinbefore described and consisting chiefly of hop bitter, so as to prepare it in a suitable form for use as a substitute for hops.
3. The treatment, substantially as set forth, of the product obtained as hereinbefore described and containing or constituting the mixture of hop resin, so as to 35 manufacture a varnish.

Dated this 17th day of February 1886.

J. C. MEWEURN.

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